Data Validation Report

TDD No: 09-04-01-0011
PAN: 001275.0440.01TA
Site: El Dorado Hills
Laboratory: Lab/Cor, Inc.
Reviewer: Denise A. Shepperd, Trillium, Inc.
Date: May 20, 2005
Revised August 11, 2005

I. Case Summary

SAMPLE INFORMATION:

Asbestos Samples:	SRA-R04-100704; SRA-R03-100704; SRA-R05-100704; SRA-R02-100704; NRA-R101-101004; NRA-R01-101004; NRA-R05-101004; NRA-R04-101004; NRA-R03-101004; and NRA-R02-101004
Matrix:	10 Air samples
Analysis:	Asbestos by Transmission Electron Microscopy
Collection Dates:	October 7 and 10, 2004
Sample Receipt Date:	October 14, 2004
Filter Preparation Date:	April 8 and 9, 2005
Grid Preparation Date:	April 11, 2005
TEM Analysis Dates:	April 18 and 19, 2005
Analytical Method:	ISO Method 13794

FIELD QC:

Field Trip Blanks (ZB):	NFB-L2-1ZB-10050; SFBB-L2-1ZB-10060; NYB-L2-1ZB-10070; JEG-L2-1ZB-10070; SRA-1ZB-1008004
Filter Blanks (FB):	SFBA-L2-FB-10050; JEG-L2-FB-101004; NRA-FB-101004
Column Balnk (FB):	NRA-FB-101005
Equipment Blanks (EB):	
Method Blank (MB):	4 glass filter blanks
Field Duplicates (D1):	Not Identified

TABLES:

1A:	Analytical Results with Qualifications
1B:	Data Qualifier Definitions for Inorganic Data Review

SAMPLING ISSUES:

No chain of custody documents were provided in the data package. These samples were originally received by the laboratory as sample lot number 041210. Chain of custody documentation that included these samples was provided on 5/4/05 by electronic transfer, at the request of the validator.

VALIDATION PARAMETERS AND COMMENTS:

I. Holding Times, Preservation and Sample Integrity

This parameter is evaluated to ensure that sample custody is documented from collection through analysis, samples are analyzed within the recommended holding time, and that no alteration in sample content has occurred during sample shipment, handling, and storage.

There is no established holding time or storage condition for asbestos samples.

II. Calibration

The analyses of materials of known content ensures that identification and quantitation of analytes will be accurate for all samples. Review of the documentation provided for appropriate calibration determines whether or not the analytical results reported by the laboratory are valid and supported by the data.

The data deliverables for this project were included in multiple data packages in several shipments. Instrument calibration documentation was provided in a separate data package in association with the site sample data packages in this shipment and included camera and screen magnification calibration (performed 4/7/05 to 4/13/05), camera length and constant calibration (performed 4/5/05 to 4/14/05), EDS peak resolution check (performed on 4/7/05), and maintenance logs for both instruments used for analyses, covering the months of March and April of 2005.

A form entitled "Microscope Based Quality Control" for the month of April 2005 was included. This form listed the various instrument calibration parameters, required frequencies, dates performed, and results. According to this form, k-factor calibration was performed most recently on the two microscopes on 1/11/05 and 3/14/05; beam dose was calibrated on 2/9/05 and 3/18/05.

No documentation of grid opening size was provided.

Documentation to support the identification and quantitation in the site samples in these data packages was provided separately with a previous shipment of data packages from the same project, and included the following:

A letter representing documentation of an NVLAP laboratory site assessment conducted on 11/7/03 was included in the data package. The letter, dated 5/10/04, indicated that the laboratory met the on-site assessment requirements.

Results and evaluator notes and tables were included for an NISTIR 5351 analysis of an inter-laboratory QC sample. The laboratory's raw data were compiled and assessed by Batta Labs. Analysts were identified by initials and included two of the analysts' initials documented with this sample set. "DW" and "KM" performed these PE sample analyses, "JH" and "TM" were not represented. According to the assessor's notes, the sample included chrysotile fibers and structures and the laboratory's results were within NVLAP and NISTIR 5351acceptance limits. No raw data were provided for this QC sample in the original data package. Raw data were provided under separate cover, at the request of the validator.

Results for a New York State Department of Health Environmental Laboratory Approval Program proficiency test, conducted between 9/7/04 and 11/9/04, were also included. The proficiency samples included asbestos in air. The laboratory's results were satisfactory for all four of the air sample categories. Actinolite and amosite fiber types

were identified and counts were acceptable, according to the data sheet. No raw data were provided for this proficiency sample. Upon request, the laboratory provided raw data documenting the identification of actinolite and amosite asbestos on 1/27/05, in conjunction with the validation of a previous shipment of data packages. These data were inserted by the validator into the QC data package provided, as supporting data, with the previous shipment of data packages.

Documentation for a round-robin sample analyzed in the fall of 2004, by three separate laboratories, as part of the NVLAP requirements, was also included in the QC data package. The documentation included raw count sheets and reported results, as well as comparison with other laboratories' results. Results for all parameters were acceptable. According to the documentation, the only analyst who participated in these analyses was "DW."

Based on the fact that the laboratory demonstrated proficiency in the performance evaluation (PE) analyses performed in the third quarter of 2004, and that these PE samples included the two predominant asbestos types detected in this field sample set, no action was taken by the validator.

III. Blanks

Sample matrices known to be devoid of the analytes of interest (method blanks) are prepared and analyzed with each analytical batch. Evaluation of this parameter ensures that contamination introduced during preparation and analyses is not attributed to the field samples.

Other blanks may be generated in the field or laboratory to ensure that no contamination is introduced during sampling and/or storage.

No field-generated blanks were included with this data set.

The laboratory preparation list and log indicated that one laboratory preparation filter blank was prepared and analyzed by the indirect method in association with the entire 70-sample set (041172, 041188, 041191, 141210, and 050680). No raw data were supplied for this blank, but a value of "0" structures detected was present on the QC summary form. According to the prep sheets, this blank was prepared on 1/13/05 (ashed and "hydrolyzed") and 3/16/05 (grid preparation). This blank would be an indicator of any contamination present in the blank filters used for preparation and for any contamination present in the prep lab environment on 1/13/05 and 3/16/05, but it does not address contamination issues for the dates on which the samples in this data set (050680) were prepared.

The indirect method used (ISO 13794), recommends analyzing a filter blank and a beaker blank with each set of samples prepared. In addition, it is recommended that the laboratory include an unused filter for preparation with every microscope slide containing sample filters that is prepared. None of these blanks were documented with this data set. Without blanks it is not possible to assess the presence or absence of laboratory contamination or its impact on the site sample results.

The laboratory had prepared blanks with the preparation and analyses of samples in this data set. At the client's request the laboratory analyzed a percentage of the prepared blanks. Data for these blanks were received by the validator on July 6, 2005. Four filter blanks, five field trip blanks, four glass filter blanks, and one column blank were prepared and analyzed as laboratory blanks. No asbestos structures were detected in any of the blanks.

IV. Spiked Samples

The analytes of interest are added in known concentrations to like-matrix blanks or authentic field samples before preparation. This parameter is evaluated in order to assess the laboratory's ability to preserve and recover the compounds of interest.

The analytical method does not require laboratory spiked sample analyses. It is recommended by the validator that some type of laboratory prepared or purchased spiked analyses be performed with each analytical sample batch.

The project requirements specified that results from the most recent inter-laboratory study would be acceptable as a laboratory control sample (LCS) for these data. This requirement was met by the laboratory and results, reported with previous data submissions for this project, for the inter-laboratory study sample were acceptable for all air sample parameters (see Section I). The data user should note, however, that no reference material was prepared and analyzed by the indirect method (ISO 13794) employed for the preparation and analyses of the site samples. This method is used when filter loading is too high to allow the use of the direct method (ISO 10312) and depends on recovery of material from the air-sampling filter, resuspension in solution, effecting a dilution, and redistribution on a secondary filter which is then prepared as for the direct TEM analysis method. Because there are additional steps involved in the preparation, there are additional error factors introduced. It is recommended that a reference material be prepared and analyzed by the indirect method so that performance can be tracked by the laboratory for support of field sample analyses by this procedure.

V. Duplicate/Replicate Samples

Results for duplicate/replicate samples are evaluated to assess the laboratory's precision for the analytes of interest in the applicable sample matrix. For asbestos analyses, duplicate and replicate measurements take the form of a combination of variables which include the preparation of the grid, the choice of grid openings to be analyzed, and the analyst performing the counting and identification of structures. For the indirect method the variables should also include preparation of the filter itself.

The laboratory included all of the QC samples from all of the field sample sets in this shipment in a separate data package under a separate report number (050580).

Two analysts, JH and TM, not represented in the PE sample analyses included with the data packages for this project, did perform intra-laboratory replicate and duplicate analyses on associated field samples. Results for these QC analyses for these analysts were within the sample-specific acceptance limits stipulated by the method.

The quality assurance project plan (QAPP) requires five types of laboratory duplicate/replicate analyses, each to be performed at a rate of 5% (one for every twenty) of the field samples. Based on the total of samples prepared by the indirect method and included in all of the data packages in this shipment combined (70 samples), four or more of each of these QC sample pairs were required (a total of 20 QC sample pairs).

The laboratory compared the primary asbestos structure count for each of the QC samples prepared and analyzed. Results for all of the duplicate/replicate pair types were evaluated based on 95% confidence limits determined from the original sample count result. Results for all of the reported QC samples were within the laboratory's calculated limits.

None of the samples in this data set (050680) was prepared as a QC pair. The laboratory analyzed a total of 11 QC sample pairs from three of the five associated data sets. A summary of the laboratory QC samples analyzed is as follows:

Replicate analyses:

- Two samples were analyzed as replicates, wherein a different preparation was analyzed by the same analyst;

Duplicate analyses:

- Three samples were analyzed as duplicates, wherein the same grid openings were recounted by a different analyst;
- Three samples were analyzed as duplicates, wherein different grid openings were selected for counting by a different analyst;
- Three samples were analyzed as duplicates, wherein a different analyst counted a different preparation.

No samples were analyzed as replicates, wherein the same analyst re-counts the same sample a second time counting different grid openings.

According to the preparation list and log, two samples (from 041188 and 041191) were re-prepared on 3/29/05 and 3/30/05. The second preparation of these two samples involved ashing the filters for two hours, according to notes on the prep list. It was assumed by the validator that these were the only samples which were carried through all of the filtering and grid preparation steps. Other QC samples listed as repreps were assumed to be additional grid preparations from the same filter only.

Agreement between the results for the 11 sample QC pairs analyzed in conjunction with the combined project-related laboratory batches in four of the required categories were acceptable. In addition, four samples were re-analyzed by the same analyst counting the same grid openings. These results were also acceptable according to the laboratory-specified limits. This category was not included as a requirement in the project QAPP.

The data user is cautioned that although the laboratory QC counts met the specified criteria, the acceptance range includes as much as a three-fold difference in asbestos concentrations for these samples. This range of variability is applicable to all asbestos results in this data set.

According to the QAPP provided with the data packages, field duplicates were required at a rate of 10% of field samples. Field duplicate pairs were not identified or evaluated as part of this validation effort.

VI. Identification

Identification of asbestos structures and fibers is dependent on sample preparation techniques, analyst training, instrument operation, and data interpretation. Comparison with results from known standards is used to evaluate the accuracy of the structure identification for field samples.

Actinolite, chrysotile, and winchite were identified in the field and QC samples. According to the report forms provided in the QC package included with a previous data package shipment for the project, the laboratory correctly identified actinolite, chrysotile, and amosite in PE sample analyses performed in the third quarter of 2004. Comparison of identification between the various analysts, grid opening, and preparations combinations that make up the daily QC for these analyses, included separately with this shipment of data packages, were within acceptance limits. Therefore; based on the documentation provided, fiber and structure identifications for chrysotile and actinolite were determined to be valid as reported. The identification of winchite was supported with EDS confirmation, however, no documentation of a reference material for this mineral was included.

VII. Quantitation and Reported Detection Limits

Raw data documentation is reviewed to ensure that all reported results and detection limits are correctly calculated, accurately reported, and supported by the raw data.

Results for asbestos categories, fiber density, and detection limits were correctly calculated and accurately reported by the laboratory. Results were verified by the validator using the information included on the reporting forms and the chain of custody records.

VIII. System Performance

This parameter is evaluated to ensure that the laboratory analytical systems were functioning properly at the time of analyses and that methodology appropriate to the analyses were followed.

The analytical systems appear to have been working satisfactorily and to have been calibrated properly at the time of these analyses, based on the documentation provided in this data package shipment. Grid opening calibration and spot size calibration were not documented

IX. Documentation

Data and documentation completeness is critical in providing support for the reported results. Problems encountered with the nature or quality of the data package documentation are addressed.

The volume of air filtered for sample NRA-R01-101004 was recorded on the COC record and the laboratory prep log as 5186.26. The volume listed on the Form I for this sample was 5186.25. This number was corrected by the validator to agree with the raw data. No adjustments to structure concentrations were necessary.

No raw data were provided in the data package for the proficiency samples analyzed in support of the laboratory's accreditation. Raw data to support the identification of actinolite and amosite were received upon request on 1/26/05 in conjunction with validation of a previous shipment of data from the same project.

Raw data for chrysotile fibers identified in only selected field samples from this data set were provided. A listing of fiber verifications was provided and included at least one structure from each of the samples in which chrysotile was identified.

Count sheets included in the data package are computer generated forms. No date of the actual count is presented on these forms. If there is a corresponding bench sheet from which these forms are prepared, these should be supplied as a part of the data package. It is recommended that analyst's intials and date of count be added to the documentation.

The legend for the count sheets, which defines the codes used for the structure counts lists PSCH as the code for protocol chrysotile structures. The code appearing on the count sheets for this category is PCAS.

COMMENTS:

These samples were analyzed by both the direct (ISO 10312) and indirect (ISO 13794) methods in order to provide comparison between the methods. In making the comparison the data user should note that the ten samples chosen had very low counts in the direct method are not appropriate for analysis by the indirect method, which is designed to accommodate the analysis of highly loaded filters. Therefore, criteria for comparison between the paired results should be chosen carefully to avoid the bias inherent in 95% confidence limits at very low concentrations. Samples with much higher structure counts would be a better choice for comparison of these two methods.

A. The volume of air filtered for sample NRA-R01-101004 was recorded on the COC record and the laboratory prep log as 5186.26. The volume listed on the Form I for this sample was 5186.25. This number was corrected by the validator to agree with the raw data. No adjustments to structure concentrations was necessary.

The laboratory reported results, analytical sensitivity, and detection limits to three significant figures. The data user should be aware that because all of these values are based on the counting of whole asbestos structures, the appropriate number of significant figures will be limited by the structure count. A total count of eight fibers will warrant results with only one significant figure; a count of 12 will warrant two, etc. Because the analytical sensitivity and detection limit are calculated from an assumed single asbestos structure, only one significant figure is accurate for these values, rather than the three reported by the laboratory. A second significant figure, if used, is considered estimated. The validator-calculated results, analytical sensitivities, and detection limits varied from the laboratory values in many cases, however, these discrepancies appeared to be due to rounding. The validator did not adjust the laboratory results to reduce the number of significant figures.

It is recommended that complete instrument calibration documentation be provided with every data package to fully support the site sample results.

The data results tables included as Table 1A include only the primary and total asbestos structure counts. Counts for individual categories required by the project Scope of Work are presented in the associated electronic data deliverables (EDD) tables.

This report was prepared according to the specifications of the analytical method, ISO Method 13794 " Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method," the document "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 10/99, and Trillium, Inc.'s SOP No. 0497-06A, for Validation of Analytical Data: Inorganic Analytes.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared in accordance with the document, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," 2/94.

U The analyte was analyzed for, but was not detected above the level of the reported value. The reported value is either the sample quantitation limit or the sample detection limit.

L Indicates results which fall between the sample detection limit and the CRDL. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.

J The associated value is an estimated quantity. The analyte was analyzed for and was positively identified, but the reported numerical value may not be consistent with the amount actually present in the environmental sample.

R The data are unusable. The analyte was analyzed for, but the presence or absence of the analyte cannot be verified.

UJ A combination of the “U” and “J” qualifier. The analyte was analyzed for but was not detected. The reported value is an estimate and may be inaccurate or imprecise.